

# Analysis of Oxidized and Reduced Compounds in the River Leith

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**Hypothesis:** An oxidizing environment, encourages the formation of an oxidized form of nitrogen (nitrate) and discourages the formation of reduced species like iron Fe(II) compounds. The opposite is true in a reducing (oxygen deficit) environment. Water and sediment samples collected from the River Leith located in England should exhibit the same behavior.

## Methods and Material:

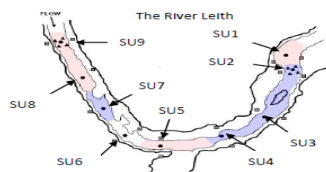


Figure 1: The River Leith and the 9 pizometer either pool sites (pink) or riffle sites (blue). The water flows from upstream [SU9] to downstream [SU1].

## Spectrophotometric determination of iron(II)

➤ Fe<sup>2+</sup> was analyzed using the spectrophotometer (it has distinctive absorption bands in certain regions of the spectrum) after it was bound to 0.1% o-phenanthroline as a ligand to improve selectivity. Another technique that needed was the use of titrations of mixtures to accurately measure the concentration Fe<sup>2+</sup>.

➤ The procedure was to prepare intermediate solution from stock solution 10,000 mg/L Fe solution to make 50ml of 50 mg/L Fe solution. Next, to prepare a calibration standard of Fe<sup>2+</sup> and each standard needed, the following reagents were added before dilution to 100ml:

- ✓ 4mL of Hydroxylammonium hydrochloride solution
- ✓ 7mL of sodium acetate buffer
- ✓ 2mL of 0.1% o-phenanthroline
- ✓ The required amount of Fe intermediate solution

Standard #	[Fe(II)] (mg/L)	Final Volume (mL)	Volume of intermediate (mL)
1	0	100	0
2	0.05	100	0.5
3	0.1	100	1.0
4	0.5	100	5.0
5	1.0	100	10.0

➤ The absorbance was measured for each calibration standard in triplicate at a wavelength of 510nm. (Make sure to zero the spectrophotometer standard 1). A calibration curve was then constructed by using a trendline to see how well it matched to the standard as well as previous calibration curves. (Detection range of concentration must be between 0-2.0 mg/L)

➤ The instrument used to identify species such as NO<sub>3</sub><sup>-</sup> and O<sub>2</sub> was the Dionex high performance liquid chromatograph. This instrument is programmed to be a time and self-regenerating instrument that was useful for sediment-free samples. The Dionex HPLC was typically used for chloride analysis. Other species with very low detection levels and small sample sizes were analyzed by a Skalar continuous flow analyzer. This instrument was generally used for the analysis of nitrates, phosphates, nitrites, oxygen, sulphates and ammonium. It also uses absorption to identify and quantify pollutants, but works well for species that are sedimentary samples. This instrument focused more upon the denitrification process.

## Data and Analysis (July 2010):

Nit... Nit... Nit... Nit...

0 0 0 0

Nit... Nit... Nit...

0 0 0

0.0  
Ox... Ox... Ox... Ox...  
0 0

Ox... Ox... Ox...

0 0

Fe(...) Fe(...) Fe(...) Fe(...)

0. 0. 0.

Fe(...) Fe(...) Fe(...)

0. 0. 0.

Data analysis of SU sites Oxidized vs. Reduced Environment			
Results	O <sub>2</sub>	NO <sub>3</sub> <sup>-</sup>	Fe <sup>2+</sup>
SU1	↑	↓	↓ then ↔
SU3	↓	↑	↓ then ↔
SU4	↓	↑	↓
SU5	↑	↔	↓
SU6	↑	↑	↑
SU7	↔	↔	↔
SU8	↑	↔	↔

↑ = increase in concentration  
↓ = decrease in concentration  
↔ = consistent concentration  
↑, ↓, ↔ = inconsistent w/ hypothesis

## Literature References:

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